U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

Technical News

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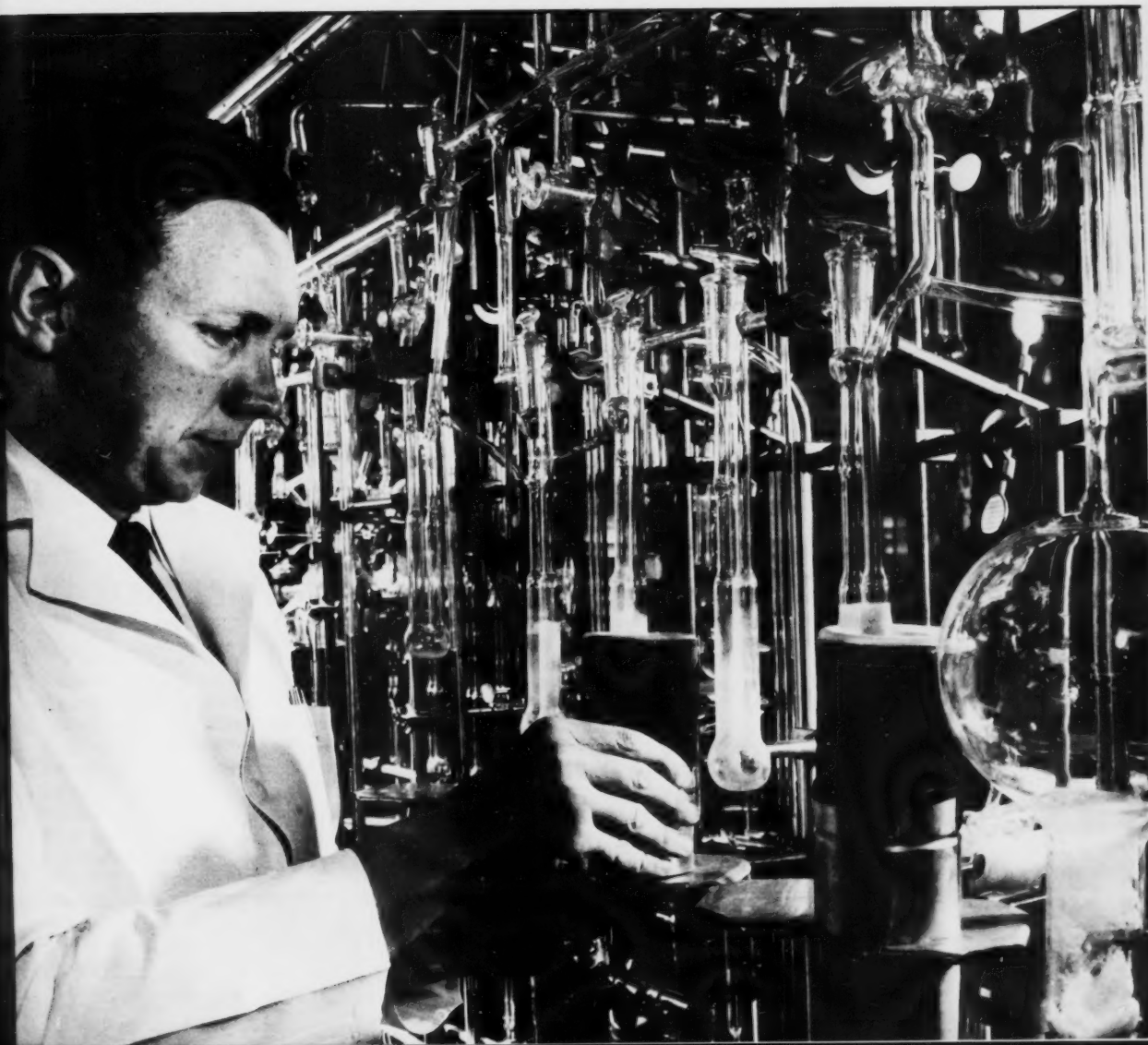

U.S. DEPARTMENT OF COMMERCE — LUTHER H. HODGES, *Secretary*

NATIONAL BUREAU OF STANDARDS — A. V. ASTIN, *Director*

NATIONAL BUREAU OF STANDARDS

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Contents

COVER: T. D. Coyle submerges a trap in liquid nitrogen while purifying difluoroborane, a new compound recently discovered at the National Bureau of Standards. Difluoroborane and other reaction products are contained in the large vessel (*foreground*). HBF_2 is released from the vessel and is purified by repeated condensations in the traps (see p. 87).

NBS chemists synthesize

FLUORINATED BORON HYDRIDE

THE FIRST example of a partially fluorinated neutral boron hydride has been synthesized at the Bureau. The new compound, difluoroborane (HBF_2), has been reported by T. D. Coyle, J. J. Ritter, and T. C. Farrar.[1] Difluoroborane was discovered during investigations of boron derivatives and will be of interest to chemists who are preparing and studying boron compounds.

Interest in the descriptive and physical chemistry of boron hydrides has been widespread in recent years from a theoretical standpoint and because they are useful as reducing agents in organic and inorganic chemistry and potentially useful as chemical fuels or fuel additives. A few boron hydrides are known in which one or more hydrogen atoms have been replaced by halogens. Prior to the work carried out at the Bureau, however, no fluorine derivatives of this type had been prepared. Difluoroborane and related compounds provide a model system useful in studying the effect of substitution on the structure and reactivity of boron hydrides. Availability of HBF_2 in quantity will permit detailed investigations of complex reactions believed to involve fluorinated hydrides as short-lived intermediates.

Two distinct methods were found for synthesizing difluoroborane. The most direct approach is pyrolysis of diborane at 100 °C in the presence of boron trifluoride. Much of the diborane is destroyed in this process, but some interchange of fluorine and hydrogen atoms also occurs, leading to the formation of HBF_2. The other, more useful synthetic procedure is the reaction of boron trifluoride with dimethoxyborane, $HB(OCH_3)_2$. The reaction is rapid, even at 0 °C. Studies of this system using isotopically labeled starting materials reveals that the reaction is complex, involving rapid exchanges of all groups bonded to boron atoms.

Difluoroborane, a gas at room temperature, boils at about −100 °C. Because it is sensitive to traces of air and moisture, high-vacuum techniques are required for its preparation and study. The compound was characterized as monomeric HBF_2 on the basis of molecular weight, infrared spectral data, and chemical reactions. The structure has been confirmed by low-temperature, high-resolution nuclear magnetic resonance studies. Its chemical properties include reaction with Lewis bases such as amines and ethers. Addition of the B—H linkage across olefinic double bonds results in formation of saturated organoboron difluorides.

HBF_2 shows several significant differences in chemical behavior when compared to the previously known chlorine analog, $HBCl_2$. For example, addition reactions of the fluoride with olefins appear to occur more slowly than the addition of the chloride. The direction of addition to some unsymmetrical olefins also appears to be different for the two compounds.

Difluoroborane and compounds related to it are being investigated intensively from a number of points of view, including descriptive chemistry and reaction mechanisms. Work at the Bureau is also being extended into the synthesis of other fluorinated hydrides and the reactions and spectroscopic properties of this class of compounds. Other investigations emphasize experimental and theoretical studies of the nuclear magnetic resonance spectra. Samples of the new compound have been provided to other groups at the Bureau for detailed spectroscopic studies.

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[1] Preparation and properties of difluoroborane, *by T. D. Coyle, J. J. Ritter, and T. C. Farrer, Proc. Chem. Soc.* **25** *(1964).*

NBS Studies Satellite Refrigerator Systems

THE NBS Cryogenic Engineering Laboratory has begun a study of refrigeration systems for satellites at the request of the National Aeronautics and Space Administration. D. Chelton, of the Institute's Boulder (Colo.) Cryogenics Division, heads the investigation aimed at improving the performance of satellite-borne infrared sensors by cryogenic cooling. Such sensors are employed in weather-forecasting satellites to determine cloud cover over the dark side of the earth.

Specifications for the satellite refrigerator call for a unit that will run for at least 3 yr without attention. The refrigerator must also conform to stringent size and weight limitations and, under present requirements, must be able to maintain temperatures of approximately 80 °K.

Some aspects of the refrigerator design will require additional research and development, but no major problems are expected. In fact, some environmental conditions of space, such as low pressures surrounding the satellite, should simplify the design.

Several interesting design ideas are already being examined. At the conclusion of the study, the Institute will present NASA a summary of some systems that might be applicable along with a definition of areas requiring further research.

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PHOTO-OXIDATION IN ASPHALTS

Studied by Infrared Spectroscopy

A STUDY of changes in the infrared spectra of asphalts due to photo-oxidation, particularly those involving carbonyl compounds with absorbance in the region of 1700 cm^{-1}, was completed recently at the Bureau. The results of this study [1] by P. G. Campbell and J. R. Wright show that, contrary to previous opinion, ketones, aldehydes, and acids, rather than esters, are the primary sources of oxygen-containing molecules in oxidized asphalt.

This study is part of a continuing program of asphalt research that the Bureau has undertaken to evaluate the quality of asphalts used in the building industry. The changes produced in asphalt by photo-oxidation are studied because photo-oxidation is the main chemical reaction in the degradation of asphalts. By determining the rate of photo-oxidation, an indication of the durability of the asphalt is obtained. Further, photo-oxidation rates provide a basis for the study of specific environmental effects on asphalt. By determining the compounds formed by photo-oxidation, effective additives may ultimately be determined that will retard or prevent photo-oxidation.

Although infrared spectroscopy has been employed to analyze asphaltene-type materials for the last 10 years, the complexity of asphalts limits the interpretation of their spectra. Similarly, the changes which occur in the infrared spectrum of an asphalt due to photo-oxidation are subject to limited interpretation. However, in the carbonyl band in the region of 1700 cm^{-1}, relative changes in the spectra of the asphalt films can be measured with considerable accuracy, even though the source of the carbonyl group cannot be easily established.

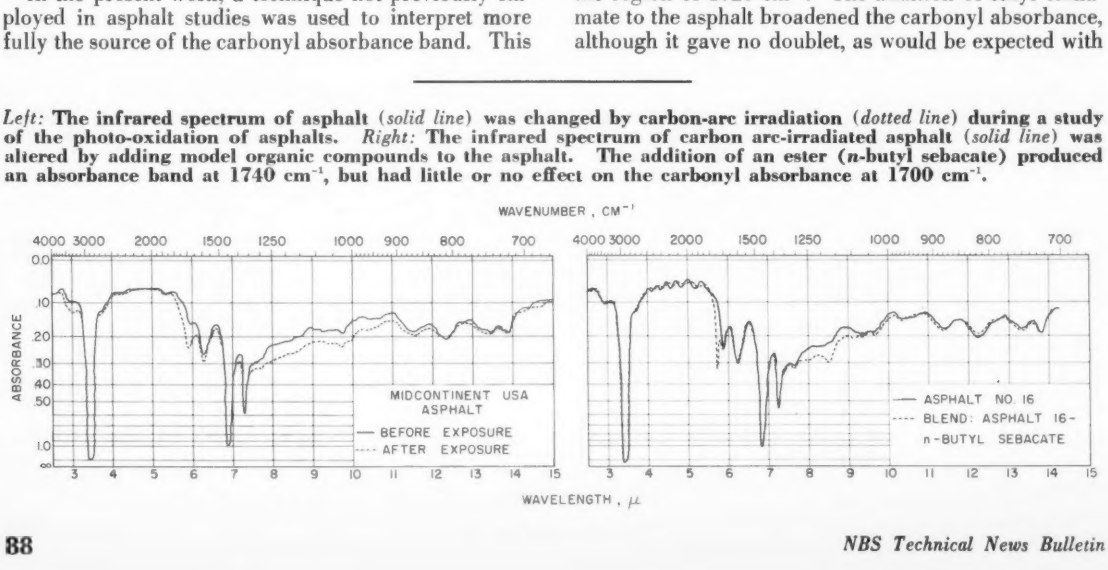
In the present work, a technique not previously employed in asphalt studies was used to interpret more fully the source of the carbonyl absorbance band. This technique employed the addition of one of 14 different model organic compounds [2] to an asphalt, the preparation of a thin asphalt film, and infrared analysis of the film. The changes that occurred in the infrared spectrum were compared with the spectrum of the untreated asphalt. New absorbance peaks or shoulders were produced if the additive was a type not present in the asphalt; existing peaks were reinforced if the additive corresponded to a compound already present.

The changes produced in the infrared spectra by the additives showed that aldehydes, ketones, and acids reinforced the carbonyl absorbance at 1700 cm^{-1}, whereas anhydrides and esters did not. The anhydrides exhibited two carbonyl absorbance bands at 1845 and 1775 cm^{-1}. The esters developed absorbance in the 1750 to 1735 cm^{-1} region, forming a doublet with the carbonyl absorbance in the asphalt. Thus, it appears that the increased absorbance found in oxidized asphalts is due largely to aldehydes, ketones, and acids, with no contribution from anhydrides and little or no contribution from esters.

Previous investigators [3, 4] attributed asphalt carbonyl absorbance in the region of 1700 cm^{-1} to esters. This contention was based on saponification values and on spectroscopic evidence (disappearance of strong carbonyl absorbance at 1724 cm^{-1} and the formation of a new band at 1587 cm^{-1} upon treatment with excess alkali, and no observation of hydrogen bridges between the solvent used in the spectroscopic method and the acids that may possibly be present).

The results of the present study do not rule out the formation of esters entirely. It is well known that α-β-unsaturated esters exhibit carbonyl absorbance in the region of 1720 cm^{-1}. The addition of ethyl cinnamate to the asphalt broadened the carbonyl absorbance, although it gave no doublet, as would be expected with

Left: **The infrared spectrum of asphalt** (*solid line*) **was changed by carbon-arc irradiation** (*dotted line*) **during a study of the photo-oxidation of asphalts.** *Right:* **The infrared spectrum of carbon arc-irradiated asphalt** (*solid line*) **was altered by adding model organic compounds to the asphalt. The addition of an ester (*n*-butyl sebacate) produced an absorbance band at 1740 cm^{-1}, but had little or no effect on the carbonyl absorbance at 1700 cm^{-1}.**



normal saturated esters. Also, in photo-oxidation of asphalts, poorly defined shoulders are sometimes observed on the main carbonyl absorbance region; these might be attributed to ester formation, since asphalts contain both acids and alcohols. However, the study does indicate that the oxygen in oxidized asphalts is principally in the forms of aldehydes, ketones, and acids (the present results do not distinguish among the classes), with a relatively minor part as esters.

[1] Infrared spectra of asphalts: some aspects of the changes caused by photo-oxidation, *by P. G. Campbell and J. R. Wright, J. Res. NBS* **68C** *(Eng. & Instr.), No. 2 (in press); Div. of Petroleum Chem. (ACS), preprint (Apr. 1964).*

[2] *n-Butyl phthalate, phthalic anhydride, ethyl cinnamate, trans-cinnamaldehyde, tristearin, stearic acid, benzoic acid, benzaldehyde, n-butyl sebacate, citral, n-heptaldehyde, acetophenone, mesityl oxide, maleic anhydride.*

[3] *J. Knotnerus, I. Inst. Petrol.* **42**, *355 (1956).*

[4] *J. M. Goppel and J. Knotnerus, Proc. Fourth World Petrol. Congr., SEc III/G, 399 (1955).*

Recalibration of the NBS Photoneutron Standard

THE BUREAU has recalibrated its standard photoneutron source with an indirect heavy-water technique, reducing the uncertainty in the result from ±2 to ±1 percent.[1] This redetermination, performed by R. H. Noyce, E. R. Mosburg, Jr., S. B. Garfinkel, and R. S. Caswell, gave an emission rate of $1.25_2 \times 10^6$ neutrons per second (n/s). The new value, combined with the weighted results of two previous determinations, gives a best value of $1.25_7 \times 10^6$ n/s.

The standard photoneutron source consists of an aluminum-covered beryllium sphere, inside of which is a cylindrical platinum-iridium capsule containing 1 g of radium in the form of radium bromide. Neutrons are released from the beryllium through the action of gamma rays from the radium. The platinum-iridium capsule blocks the outward passage of alpha particles from the radium, preventing unwanted alpha-neutron reactions. This source provides the basis for the neutron-source calibration services offered by the Bureau.[2]

The standard had been calibrated twice before. In the first determination[3] the volume integral of the absorption rate in light water was measured with indium and manganese foils. This technique has an uncertainty of ±3 percent. A second determination, having an uncertainty of ±2 percent, was made by measuring the gamma-ray activity induced in manganese 55 through the capture of neutrons.[4] A light-water bath of manganous sulfate was used, and the gamma-ray activity was determined with a Geiger-Müller counter suspended within the bath.

As errors are introduced in the results of the earlier calibrations by uncertainties in the cross sections of various elements, it was decided to recalibrate the standard in a heavy-water solution of manganous sulfate. The advantage of using heavy water in the calibration is that deuterium has a much smaller likelihood of capturing neutrons than does hydrogen, thus reducing the error arising from uncertainties in the capture cross sections. The standard source could not, however, be calibrated directly in the heavy-water bath. This is because some of the gamma rays coming from the source (from the decay of radium) are energetic enough to cause the photodisintegration of deuterium, creating a background of neutrons nearly equal to the neutron yield of the source. Therefore, an antimony-beryllium (Sb-Be) source, which has few gamma rays above the photodisintegration threshold of deuterium, was calibrated in the heavy-water manganous sulfate bath, and then compared with the standard source in a light-water manganous bath.

Before the emission rate of the Sb-Be source could be determined in the heavy-water bath, the efficiency of the dip scintillation counter and bath had to be established. This was done by adding a known amount of manganese 56 to the bath and determining the gamma-ray count rate. The manganese 56 was produced by irradiating a solution of manganous sulfate in the University of Maryland reactor. The solution was then diluted to 25 ml in 0.5 *N* HCl, and portions ranging from 0.03 to 0.10 ml were withdrawn and measured by $4\pi\beta$-γ coincidence counting. The mean activity of the solution was determined to be $100.5_8 \times 10^3$ disintegrations per second per milliliter. The solution remaining after withdrawal of the samples was mixed into the heavy-water bath, and the count rate determined. From the count rate resulting from the addition of this known amount of activity, the efficiency of the counting system was established as $6.69_6 \times 10^{-5}$ counts per disintegration of manganese 56.

The emission rate of the Sb-Be source was then determined. The source, contained within a carbon block and suspended by stainless steel wires, was allowed to remain within the bath for about 18 hr. After the source was removed from the bath, the dip counter

Disassembled replica (4 cm in diam) of the national neutron standard maintained by the Bureau. Neutrons are produced through the action of gamma rays, coming from radium within the platinum-iridium cylinder, on the outer beryllium sphere. The source was recently recalibrated by an indirect heavy-water technique, a value of $1.25_2 \times 10^6$ n/s being found.

was inserted and the gamma-ray activity determined. This procedure was repeated 8 times, the corrected emission rate of the source being $0.276_0 \times 10^6$ n/s. Such factors as loss of neutrons from the bath; absorption of neutrons by the source, carbon block, and suspension wires; photodisintegration of some of the deuterons; evaporation; residual activity from the previous calibration; and others had to be considered in the final calculations.

Once the emission rate of the Sb-Be source was known, operations were transferred to a light-water manganous sulfate bath. Here, the source was immersed much as in the heavy-water bath, and the gamma emission rate of the manganese 56 determined after the source was removed. Seven determinations were made with the Sb-Be source, and four with the Ra-Be standard source. From the ratio of the corrected activities determined in the light-water bath, and the known activity of the Sb-Be source as determined in the heavy-water bath, the emission rate of the standard source was calculated.

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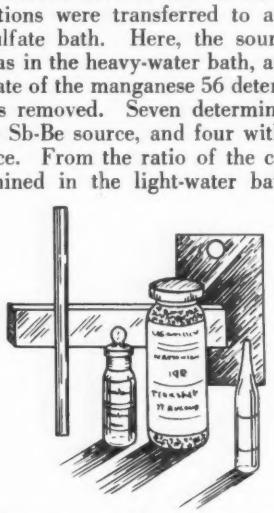
[1] *For further details, see* Absolute calibration of the NBS photoneutron source: III. Absorption in a heavy water solution of manganous sulfate, *R. H. Noyce, E. R. Mosburg, Jr., S. B. Garfinkel, and R. S. Caswell, J. Nuclear Energy, Parts A/B, 313 (Sept. 1963).*

[2] *Calibration services provided by NBS are listed in NBS Misc. Publ. 250,* Calibration and test services of the National Bureau of Standards, *available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, for 70c.*

[3] Absolute calibration of the NBS photoneutron standard: I. *J. A. DeJuren, D. W. Padgett, and L. F. Curtiss, J. Res. NBS* **55,** *63–69 (Aug. 1955).*

[4] Absolute calibration of the NBS photoneutron source: II. Absorption in manganese sulfate, *J. DeJuren and J. Chin, J. Res. NBS* **55,** *311–316 (Dec. 1955).*



STANDARD MATERIALS

Spectrochemical Standards Reevaluated for Microanalysis

WORK has begun to reevaluate several existing NBS spectrometric standard materials to determine their usefuless as standards for instrumental microanalytical techniques.[1] R. E. Michaelis, H. Yakowitz, and G. A. Moore have examined the first of these materials, a low-alloy steel known as NBS standard No. 461, for homogeneity; type, size, and shape of inclusions; and grain size. The results of this evaluation indicate that NBS standard 461 is sufficiently homogeneous to permit it to be used in any present instrumental microanalytical technique at the 5-μ level.

Most modern instruments employed for rapid analyses are calibrated with standard samples of known composition.[2] In cooperation with industrial and governmental groups, the Bureau plans, prepares, tests, and certifies selected standards of composition to serve science and technology in critical areas of calibration.[3] Standards are intended for a specific use and usually are not satisfactory for other uses. For example, steel samples issued by the Bureau in chip form for chemical analysis should not be assumed to be suitable for spectrographic analysis. Similarly, spectroscopic standard samples may not be applicable for other instrumental methods.

The Bureau has therefore begun to reevaluate some of its existing spectroscopic standards to determine their applicability as standard samples for new instrumental analytical techniques such as solids mass spectrometry, electron probe microanalysis, activation analysis, or laser methods in optical emission analysis. These techniques require standard specimens having particle size and composition homogeneity at the micron level. Such specimens must be characterized in greater detail than is necessary for standards used in normal optical emission or x-ray spectrometric analyses.

In choosing a material to begin the reevaluation program, photomicrographs of eight available NBS spectroscopic standards were examined. These studies revealed that several of the newer steel standards appeared to have structural homogeneity at the 10- to 20-μ level. Two standards (No. 461 and No. 1161) made from the same material were initially selected. Of these two cylindrical samples, which are identical except for size, the smaller (No. 461) was chosen because it had received more severe cold-working than the larger, and hence exhibited a finer grain size.

In the subsequent analysis, the NBS electron probe microanalyzer [4] was employed to determine the homogeneity of both nickel and iron at the 2-to-4-μ level of spatial resolution in the longitudinal, transverse, and normal directions. Forty-six separate spot determinations were made for nickel in the sample's 0.556 cm diameter cross section, and 62 determinations were made in a 10 cm length. For iron, 9 spot determinations were made in the sample cross section and 16 in its length. Inclusions in the samples were identified, their volume percentage calculated, the mean free path between them determined, and their number and size distributions found by using both electron probe and chemical etching techniques.

Concerning the steel itself, the following information was obtained: (a) the ferrite and pearlite per-

Harvey Yakowitz uses an electron probe microanalyzer to investigate the microstructure of NBS standard sample No. 461. Results of the investigation showed that this spectrochemical standard is sufficiently homogeneous to permit it to be used with any present method of instrumental microanalysis at the 5-μ level.

centages in the worked structure of the steel, (b) the mean free path for ferrite and pearlite, and (c) the grain size of the steel. To obtain this information, a technique of quantitative metallography [5] employing a digital computer capable of accepting suitable photomicrographs and of directly printing out the desired information was used.

With this method, high-contrast black and white photomicrographs of the specimen are scanned and the resulting data introduced into the computer in binary machine code. Twenty-eight general computer operations can be performed on each photomicrograph by feeding a master routine into a portion of the computer memory. These analyses resulted in a more complete characterization of standard 461 than had previously been available.

Work is in progress at the Bureau to determine the feasibility of using other NBS spectrochemical standards for microanalytical techniques.

[1] Metallographic characterization of an NBS spectrometric low alloy steel standard, *by R. E. Michaelis, H. Yakowitz, and G. A. Moore, J. Res. NBS* **68A** *(Phys. & Chem.), to be published.*

[2] Report on available standard samples, reference samples, and high purity materials for spectrochemical analysis, *by R. E. Michaelis, ASTM Spec. Tech. Publ. No. 58-E (1964).*

[3] *Standard materials available from NBS are listed in* Standard Materials, *Misc. Publ. 241, which may be obtained for 30 cents from the Superintendent of Documents, Washington, D.C., 20402. Up-to-date supplementary insert sheets to Misc. Publ. 241 list new, renewal, out of stock, and discontinued standards. This sheet is available without charge directly from the National Bureau of Standards, Washington, D.C., 20234.*

[4] Metallurgical microanalysis with the electron probe, *by J. R. Cuthill, L. L. Wyman, and H. Yakowitz, J. of Metals of the Metallurgical Society of AIME* **15,** *763 (Oct. 1963).*

[5] Digital computer used for quantitative metallographic analyses, *NBS Tech. News Bull.* **47,** *No. 2, 26–29 (Feb. 1963).*

NBS Issues Four New Radioactivity Standards

The NBS Institute for Basic Standards has recently issued four new radioactivity standards: hydrogen 3, strontium 89, cerium 141, and cesium-barium 137.[1] Supplies of sodium 22, manganese 54, and radium 226 standard samples, which were previously exhausted, have been replenished and are again available.

Radioactivity standards are used for calibrating equipment employed to measure the activities of other radioactive samples which emit radiation having similar characteristics. Such radionuclides have wide applications in the fields of medicine, fundamental scientific research, and industry.

NBS standard samples of strontium 89 and cerium 141 are both beta-ray solution standards of radionu-

Sample No.	Radionuclide	Form	Activity or emission rate	Half-life	Price
		New Radioactivity Standards			
4945	Strontium 89	3-ml solution	2.7×10^4 dps/ml (Dec. 5, 1963)	50.5 days	$30
4946	Cerium 141	3-ml solution	5.2×10^4 dps/ml (Jan. 3, 1964)	32.5 days	49
4200	Cesium-barium 137	Point source	5×10^4 γ/sec (Aug. 1, 1963)	~30 years	46
4947	Hydrogen 3	5-ml solution	3×10^5 dps/ml (Feb. 1964)	12.2 years	30
		Reissued Radioactivity Standards			
4997-C	Manganese 54	Point source	5.0×10^4 γ/sec (Jan. 10, 1964)	312 days	57
4229-D	Sodium 22	5-ml solution	76×10^4 β^+/sec (Jan. 15, 1964)	2.59 years	37
4950-A	Radium 226	100-ml solution	10^{-9} curie	1620 years	42

clides encountered in fallout. The materials used to prepare these standards were obtained from irradiation of enriched stable isotopes, rather than from fission-product mixtures. The resulting standard samples have a very low content of long-lived radioisotopes such as strontium-yttrium 90 and cerium-praseodymium 144.

The strontium 89 standard samples, priced at $30 for each 3-ml sample, have a nominal activity of 2.7×10^4 dps/ml (disintegrations per second per milliliter) and a half-life of 50.5 days. The cerium 141 standards, priced at $49 for 3 ml, having a nominal activity of 5.2×10^4 dps/ml and a half-life of 32.5 days.

Another beta-ray solution standard, hydrogen 3, consists of 5 ml of tritiated toluene and has a nominal activity of 3×10^5 dps/ml. Priced at $30, this material has a half-life of 12.2 yr and was developed to calibrate instruments used in liquid scintillation counting.

Cesium-barium 137 gamma-ray point-source standards are available at a price of $46. These standards, designated NBS sample No. 4200, can be used in calibrating gamma-ray spectrometers. Each standard has a nominal emission rate of 5×10^4 γ/sec (gamma rays per second) and a half-life of approximately 30 yr.

Another NBS gamma-ray point-source standard, manganese 54, with a 312-day half-life, has been reissued as NBS sample No. 4997–C. It is priced at $54, and has a nominal emission rate of 5×10^4 γ/sec.

Sodium 22 has been reissued as NBS sample No. 4922–D. This 5-ml sample, with a half-life of 2.59 yr and an emission rate of 76×10^4 β^+/sec (positrons per second), is priced at $37.

The reissued standard of radium 226, designated NBS sample No. 4950–A, has an activity of 10^{-9} curie. Issued as a 100-ml solution for $42, the radium 226 standard is used for radon analysis.

All orders for radioactivity standards should be addressed to:

Administrative Clerk
Radioactivity Standard Samples
Radioactivity Section
National Bureau of Standards
Washington, D.C. 20234.

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[1] *Standard materials issued by the National Bureau of Standards are described in* Standard Materials, *NBS Misc. Publ. 241. This publication may be ordered for 30 cents from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402. Up-to-date supplementary inserts, which are issued periodically, are available upon request directly from the National Bureau of Standards.*

Computer Predicts Living Conditions in Underground Protective Structures

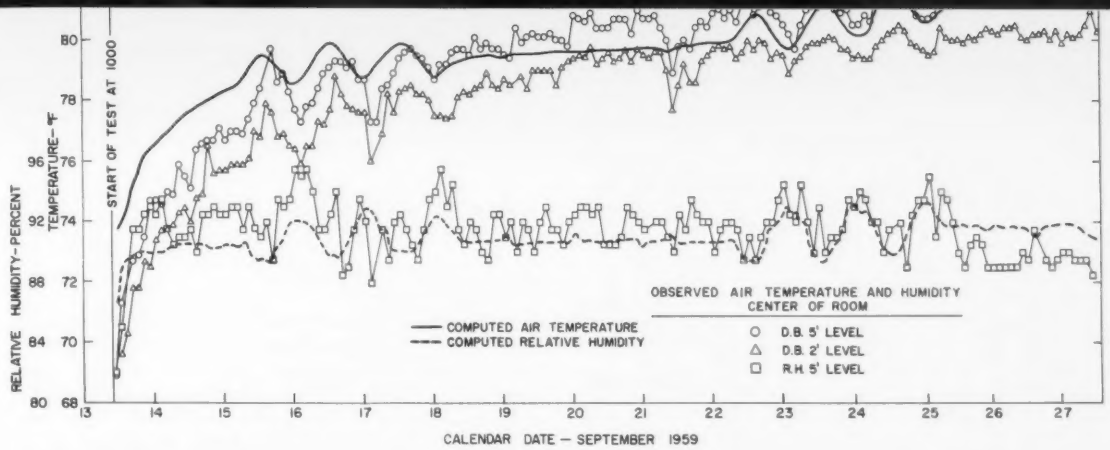
THE BUREAU, in cooperation with the Office of Civil Defense, has developed a method for predicting environmental conditions in proposed underground protective structures.[1] The method, devised by T. Kusuda and P. R. Achenbach, employs a digital computer simulation technique and gives results that compare favorably with experimental observations. Such calculations will be useful in designing and evaluating underground fallout shelters in locations where earth and climatic temperature data are available.

Some of the earliest studies of fallout shelters were made to evaluate their structural safety and fallout protection. More recent experimental investigations conducted at the Bureau [2] have indicated the importance of the thermal environment upon occupants of fallout shelters. The metabolic heat generated by the shelter occupants and heat produced by internal lighting or power systems must be dissipated either to the surrounding earth or to the ventilation air. The hour-by-hour balance between the generation of heat and its removal by transmission or ventilation determines the overall shelter temperature. Likewise, the humidity in a shelter depends on the dynamic balance between moisture evaporation and condensation and moisture removal by the ventilation air.

The present investigation was undertaken to evaluate the feasibility of using the digital computer to solve the problem of simultaneous exchange of heat and water vapor among human occupants, ventilation air, and the walls of a three-dimensional earth cavity at finite depths below the earth's surface. The earth temperature surrounding the cavity during the occupancy period was calculated by a finite difference time iteration technique. Solar radiation and ambient air heat exchange with the earth's surface over the shelter were also included in the calculation.

The Bureau's computer program can predict shelter air temperature, relative humidity, shelter inner surface temperature, and shelter inner surface heat flux at 2-hr intervals during a 14-day occupancy period. The computer results compared favorably with the data observed for the same time period in two prototype shelters. In one of these, a 6-man, family-size shelter located on the Bureau grounds, data were obtained during one winter and two summers. In the other structure, an 18-man community shelter located at the University of Florida, data were obtained during one summer.

Although finite difference solutions to differential equations are inherently subject to errors due to the finiteness of the matrix grid and to rounding off of the significant figures during iterative calculation steps, the accuracy of predicted environmental conditions for an actual shelter depends primarily on the reliability of input data. These data include thermal properties of the earth, outdoor weather conditions, initial earth temperature profile, and surface heat transfer coefficients. A major advantage of the computer technique in this application is that the number of simplifying assumptions for the calculation can be significantly decreased,



Computed values of air temperature and relative humidity superimposed on the experimental data observed during one summer test in the NBS family-size fallout shelter.

with a corresponding increase in the probable reliability of the computed results.

The mathematical relations necessary for solving the three-dimensional problem have been written in Fortran computer language and programmed into a 7090 computer. A listing of the Fortran program as well as the assembled binary card deck are available from the Bureau.

The program is being applied to other larger-size shelters for further comparison and will eventually be used to study the effects of various design parameters. Similar three-dimensional heat transfer problems in underground cavities other than fallout shelters can also be analyzed by this program with some modification of the external boundary conditions of the system.

[1] Numerical analyses of the thermal environment of occupied underground spaces with finite cover using a digital computed, *by Tamami Kusuda and P. R. Achenbach, American Society of Heating, Refrigerating and Air-Conditioning Engineers Preprint (1963).*

[2] Environmental characteristics of a small underground fallout shelter, *by P. R. Achenbach, F. J. J. Drapeau, and C. W. Phillips, ASHRAE Journal* **4,** *21 (1962). Also,* Simulated occupants aid study of family-size underground fallout shelter, *NBS Tech. News Bull.* **46,** *26–30 (Feb. 1962).*

Mathematical Technique for Evaluating Optical Systems

From Local Properties of Wave Fronts

A METHOD has been developed for determining the local properties of a wave front associated with a beam of light propagating through an optical system when only the design data of the system are known. With this method,[1] originated by John A. Kneisly II, it is possible to follow the changing shape of a wave front propagating through almost any optical instrument and to determine the geometric structure of the resulting image. For example, the method is being applied to the evaluation of a proposed design for a grating spectrometer.

In the past, the Bureau has developed various other techniques for studying the properties of optical systems when only their design specifications are known. However, in these techniques spot diagrams are used and the validity of geometrical optics is assumed. The results are sufficiently accurate as long as the geometric aberrations of the lenses under study are large compared with their diffraction effects.

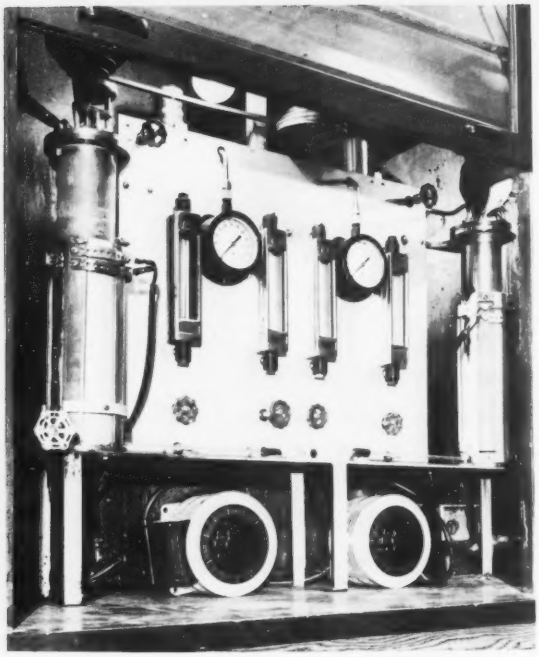
More recently, however, the design of optical systems has improved to the extent that geometric aberrations can be made very small—so small that diffraction effects assume a greater importance. Such lenses are often called "diffraction limited," and their spot diagram analysis is completely unreliable.

The present Bureau method of wave front analysis is based on the formal methods of differential geometry and makes use of ray-tracing formulas in vector form. This method enables one to determine at any point on a wave front its principal curvatures, which provide the astigmatic foci arising from the wave front at this point. In addition, the amplitude of the electric and magnetic vectors associated with the wave front at this point are related to the product of the principal curvatures. This relation opens the door to the determination of the structure of the true image—taking into account the effects of diffraction—formed by an optical system when only its design is known.

At present, the equations for this type of wave front analysis as applied to spherical refracting and reflecting surfaces as well as to plane diffraction gratings have been programmed for the Bureau's 7090 computer. Possible applications are to the design of optical systems, including grating spectrometers, photogrammetric cameras and lenses, telescopes, and optical microscopes, and to theoretical investigations into the structure of the diffraction patterns associated with optical image formation.

[1] *For further details, see* Local curvature of wavefronts in an optical system, *by John A. Kneisly II, J. Opt. Soc. Am.* **54,** *No. 2, 229–235 (Feb. 1964).*

Effect of Blowing C
Asphalt Durability a



The two blowing stills used during a study of the effects of blowing variables on the durability of asphalt (agitated still, *left*; nonagitated still, *right*) with individual rotameters (*center*) and variable transformers (*bottom center*).

A STUDY [1] of the effect of blowing variables on coating-grade asphalts has recently been completed at the Bureau. The study was conducted by Sidney H. Greenfeld, a research associate of the Asphalt Roofing Industry Bureau at NBS, to determine what effect varying temperature, air rate, and agitation conditions had on the durability of asphalts.

Blowing is the controlled oxidation of asphalt. Oxygen combines with asphalt compounds in various ways, with resultant changes in the physical properties of the asphalt. The changes that occur during blowing consist of hardening, decreasing temperature susceptibility, and shifting to a more gel-like substance. Although the blowing process has been used for almost a century to modify the properties of organic material, little or no attention has been given in other asphalt studies to the effect of blowing variables on asphalt durability. Therefore, durability was the primary concern of this study, while physical properties were of secondary interest.

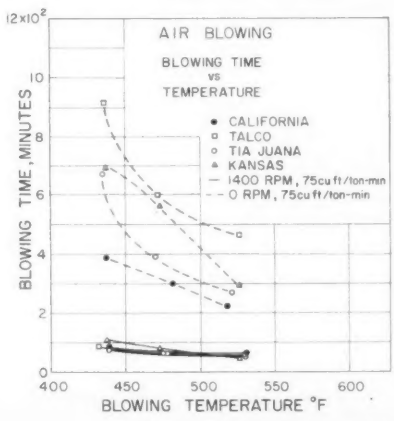
When crude oil (petroleum) is distilled, lightweight hydrocarbons are removed and condensed separately into various products such as gasoline, kerosene, and oils. After distillation is completed a residue of heavy hydrocarbons remains. This residue or asphalt flux is further processed, by blowing and other operations, to form various grades of asphalt.

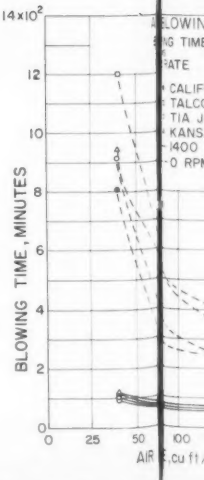
California, Kansas, Talco, and Tia Juana fluxes were chosen for the present study as representing the majority of fluxes used in asphalt production in the United States. Under varying conditions of temperature, air rate, and agitation, these fluxes were blown in two 2-liter stainless steel blowing stills to form coating-grade asphalt. All tests were run at atmospheric pressure.

Blowing conditions were the same for both stills with the exception of agitation. Temperature was varied from 430 °F to 530 °F by use of two variable transformers. Air rate, controlled by separate rotameters, varied from 38 to 150 ft^3/ton-min.

In the non-agitated still, compressed air, filtered through glass wool, was delivered through a 3/8-in. stainless steel tube to the junction of the four arms of a cross made of hollow tubing. The air passed through 12 equally spaced holes, three per arm, on the underside of the tubing into the asphalt. In the agitated still the air input tube carried the air to the bottom of the still just below the center of the first of three impellers. As air passed through the impeller blades it was divided into small bubbles which were further divided by each succeeding impeller.

Left and center: **Graphs of asphalt blowing time versus temperature and not agitated during blowing, and solid lines when the asphalt was agitated the NBS experiment show that increases in temperature and air rate greatly but have only minor effect on agitated asphalt.** *Right:* **The graph blow greatly reduce blowing time.**





ng Conditions on ity and Blowing Time

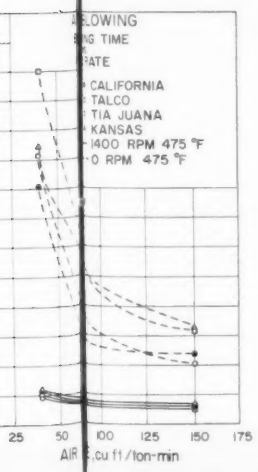
Samples of the asphalt were taken periodically for softening point determinations. Air flow was interrupted to remove the sample and started again after the sample was removed, except when the softening point was expected to exceed 200 °F. The air was left off during these samplings to prevent overblowing.

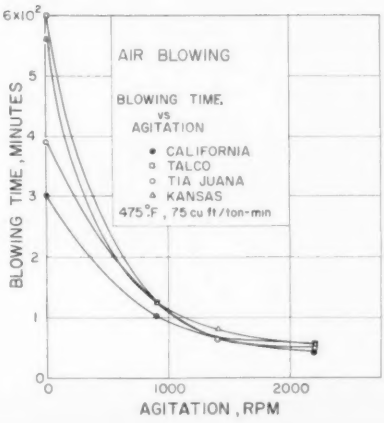
There is no general specification for asphalt performance; rather each producer has determined a narrow specification suited to his needs. Therefore, for purposes of this study a hypothetical specification, broad enough in range to cover the majority of producers' specifications, was drawn up. The hypothetical specification is shown in table 1.

TABLE 1. *Hypothetical coating-grade asphalt specification*

Property	Range
Softening Point, °F	215–225.
Penetration @ 32 °F, 1/10 mm	10 minimum.
Penetration @ 77 °F, 1/10 mm	15–25.
Penetration @ 115 °F, 1/10 mm	25–45.

us temperature and air rate are shown as dotted lines when the asphalt was asphalt was agitated at 1400 rpm during blowing. Plotted data obtained in re and air rate greatly reduce blowing time when the asphalt is not agitated, The graph blowing time versus agitation shows that increases in agitation





Each of the four fluxes was subjected to varying blowing conditions in order to produce an asphalt that met this specification. All of the Kansas and Tia Juana asphalts produced met the specification and their penetrations were essentially independent of blowing conditions. Only in low-temperature penetration did the two Talco asphalts blown at 430 °F fail to meet the specification, and they failed by only one unit. None of the California asphalts met the specification. Unlike the Kansas and Tia Juana fluxes, the California and Talco fluxes produced asphalts whose penetrations varied with blowing conditions.

Blowing time was greatly affected by the variables of the experiment. Of the four fluxes, the blowing time for Talco varied the most. The other asphalts exhibited similar variations but of lesser magnitude. In the non-agitated still, air rate was held constant at 75 ft^3/ton-min and the blowing time required varied with temperature from 15 hr at 430 °F to about 7½ hr at 530 °F. With temperature constant at 475 °F, blowing time varied with air rate from about 20 hr at 38 ft^3/ton-min to about 5 hr at 150 ft^3/ton-min.

In the agitated still with agitation held at 1400 rpm and temperature at 475 °F, blowing time varied with air rate from about 1¾ hr at 38 ft^3/ton-min to about 1 hr at 150 ft^3/ton-min. With air rate and temperature held constant at 75 ft^3/ton-min and 475 °F, respectively, blowing time varied from about 10 hr to 1 hr, as agitation varied from 0 to 2200 rpm, respectively.

The data obtained showed that increasing the temperature, air rate, and agitation will decrease blowing time. However, the most rapid blowing time did not produce the most durable asphalt. Generally, the milder blowing conditions produced the more durable asphalts, with the less durable fluxes being more sensitive to the blowing variables.

Optimum ranges of blowing conditions for producing the more durable asphalts were found for each flux. These conditions are listed in table 2.

TABLE 2. *Optimum blowing conditions for various fluxes*

Flux	Durability	Temp.	Agitation	Air rate
	days	*°F*	*rpm*	*ft^3/ton-min*
Calif.	62–68	439–483	1400–2200	75–150
Kansas	80–87	437–475	0–2200	38–75
Talco	50–56	432	0	150
Tia J	58–60	435–470	0–900	75–150

As seen in table 2, the ranges of variables for some fluxes were large, while for others the ranges were narrow. By using the maximum setting of each variable for a given flux as listed in table 2, it is possible to obtain one of the more durable asphalts from each flux in the shortest period of blowing time.

[1] *For further details,* see The effect of blowing variables on the durability of coating-grade asphalts, *by S. H. Greenfeld, Ind. Eng. Chem. Prod. Res. Develop.* **3, 2** *(June 1964).*

NONLINEAR IONOSPHERIC PROCESSES

Conference at NBS Boulder Laboratories

THE NBS BOULDER (Colo.) Laboratories was host December 16 and 17, 1963, for a Conference on Non-Linear Processes in the Ionosphere, cosponsored by the Voice of America (VOA) and the Bureau's Central Radio Propagation Laboratory (CRPL). The Conference attracted radio scientists from government, university, and industrial laboratories in the United States, Canada, Australia, and Italy. Two morning sessions were devoted to radio wave interactions involving collisions in the ionosphere. One of the afternoon sessions directed attention to collisionless radio wave interactions and the other to excitation processes in the ionosphere.

The cochairmen of the Conference were Dr. Donald H. Menzel (Harvard College Observatory) and Dr. Ernest K. Smith (CRPL); Robert T. Frost (CRPL) was secretary-treasurer. Dr. C. Gordon Little (CRPL) was chairman of the advisory committee, the other members of which were Dr. James R. Wait (CRPL), Roger M. Gallet (CRPL), Prof. David Layzer (Harvard), and George Jacobs (VOA).

The ionosphere plays a major role in long-distance radio communication and radio broadcasting because of the free electrons which it contains. Normally the radio energy incident on the ionosphere is not enough to affect its physical properties and therefore radio signals reflected from the same part of the ionosphere do not interact with one another. The scientists at the Conference exchanged information on conditions of power and frequency, etc., under which radio waves change the ionosphere, the resultant consequences in the ionosphere, and the effect on radio waves propagating through the modified region.

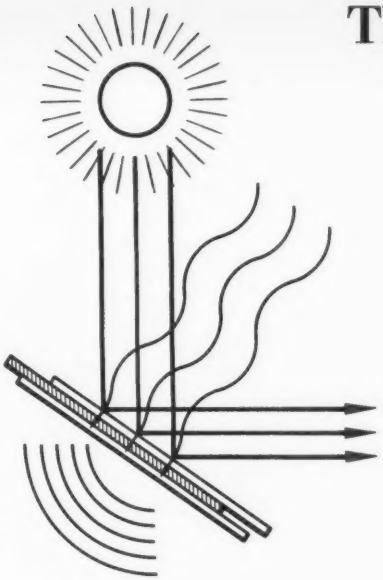
The Conference was opened with some general remarks on nonlinear processes in a session devoted to collisional radio wave interactions and presided over by Prof. Menzel. The use of the Jicamarca (Peru) Radar Observatory to investigate ionospheric reactions and to measure the Luxembourg effect was described. Data were introduced to support the conclusion that the cross-modulation reaction takes place at a height of about 75 km. The growth of awareness that strong radio waves can modify the ionosphere was described as leading to an understanding of changes in ionospheric electron temperature, collision frequency, rates of attachment and recombination, and excitation. One paper dealt with the energy maxima at the gyromagnetic resonance following elastic or inelastic collisions and components of the complex dielectric permittivity tensor.

The second session, on collisional interactions, was presided over by Prof. M. Cutolo of the University of Naples. It included the presentation of a microscopic theory of ionospheric cross modulation, the extension of magneto-ionic theory to include ion motions, its application to the propagation in the terrestrial ionosphere and the solar corona, and the use of a solution for cold plasma in treating a plasma cloud approaching a dipole, such as at the onset of a magnetic storm. Other papers described ionospheric wave interaction by gyrowaves in the nighttime lower *E*-region, wave interaction at oblique incidence near the gyrofrequency, and nonlinear propagation in magnetoplasma.

The session on collisionless radio wave interactions, held under the chairmanship of Dr. J. A. Fejer of the Southwest Center for Advanced Studies, presented surveys of natural and artificially stimulated VLF emissions. VLF signals received by the joint U.S.-Canadian Alouette I satellite were described as consisting of bands of noise related to the geomagnetic field. The observed phenomenon was compared with such theories of the generation of exospheric noise as the Doppler-shifted cyclotron and the traveling-wave tube amplification theories. Plasma resonances attributed to electrostatic oscillation of ionospheric electrons and supporting oscillation for a few milliseconds were reported to occur at the electron plasma frequency and at multiples of the electron gyrofrequency. The interaction of an antenna with a plasma and plasma instability in the whistler mode caused by a gyrating electron stream also were discussed in the session.

The final session of the Conference, held under the chairmanship of Prof. V. A. Bailey of the University of Sydney (Australia), considered excitation processes operative in the ionosphere. Changes in the ionosphere brought about by radio energy traversing it were described; papers dealt with the resulting decrease in *D*-region electron density, optical radiation (such as night airglow) resulting from gases raised to their excited states, and changes in propagation of a beam caused by electrons created by its own passage. The rapid increase in mean electron energy caused by heating of electrons in nitrogen and air by rf fields was treated. The feasibility was discussed of low-frequency emissions arising naturally in the exosphere in traveling-wave-type interactions between drifting ionized streams. The dependence of modulation impressed on partial echoes from the *D*-region was attributed not only to interaction below the reflecting discontinuity, but also to a previously unknown change in the reflective power of the discontinuity. The amplitude ratio of the right and left circularly polarized echoes was shown to depend on the orientation of the polarization plane relative to the magnetic meridian. The final paper described nonlinear effects resulting from passing electron beams through refractive gratings and attempted to explain the phenomena by regarding the beam as a plasma.

The Proceedings of the Conference on *Non-Linear Processes in the Ionosphere* are being published as NBS Technical Note 211, edited by E. K. Smith and D. H. Menzel. The six volumes comprising this Technical Note consist of 664 pages containing 20 of the papers presented at the Conference. It is obtainable from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402 by the individual volumes and at $3.35 for the complete set.



Thermal Radiation Symposium Held in San Francisco

A Symposium on the Thermal Radiation Properties of Solids was held March 4–6, 1964, at the Sheraton-Palace Hotel in San Francisco, Calif.[1, 2] The symposium was sponsored jointly by NBS, the National Aeronautics and Space Administration (NASA), and the Research and Technology Division of the U.S. Air Force (USAF). Host for the meeting was the University of California at Berkeley. The meeting's general chairman was J. C. Richmond of NBS. Assisting him were J. H. Charlesworth, USAF; J. J. Gangler, NASA; G. B. Heller, NASA; and R. A. Winn, USAF.

Over 300 scientists and engineers, representing U.S. Government agencies, American industry, and several foreign countries, attended the Symposium. Thirty-three scientific papers were presented in four technical sessions. A fifth session was devoted to a panel discussion of the role that surface effects play in modifying radiative properties of materials.

The meeting opened with a welcoming address by H. J. Allen, Assistant Director for Astronautics, Ames Research Center, NASA. Mr. Allen discussed the application of thermal radiation properties in modern scientific and space engineering problems. A brief response to his address was given by E. P. Wheaton, Vice President and General Manager, Research and Development Division, Lockheed Missiles and Space Company.

Highlighting the Symposium was the banquet meeting, between the fourth and fifth sessions. Guest speaker, Dr. E. U. Condon, Professor of Physics at the University of Colorado, and former Director of the National Bureau of Standards, spoke of the need to exchange scientific information more effectively. He commended the Symposium participants for their essential work which directly affects this nation's space program.

Session I

The first technical session, "Fundamentals," was presided over by H. H. Blau, Jr., of Arthur D. Little, Inc., Cambridge, Mass. The opening paper, presented by A. G. Emslie, of Arthur D. Little, Inc., defined problem areas associated with thermal radiation theory. Such problems involve calculations, statistical considerations, and computational complexities rather than principles.

The next presentation, by W. J. Parker and G. L. Abbott, of the U.S. Naval Radiological Defense Laboratory (USNRDL), San Francisco, Calif., elucidated some of the mathematical concepts for such general expressions as total normal and total hemispherical emittance. They described total emittance measurements they had made on several metals and compared their experimental observations with theoretical data in this field.

J. R. Aronson and H. G. McLinden, of Arthur D. Little, Inc., spoke of their work on the radiation properties of solids in the far infrared region of the spectrum. Many of their investigations consisted of measurements made at temperatures of 25, 50, 75, 100 °K in the spectral range of 50 to 500 cm^{-1}.

Equations and curves for the ratios of hemispherical emissivity to normal emissivity, which are obtained from electromagnetic theory, were reviewed by R. V. Dunkle, of the Commonwealth Scientific and Industrial Research Organization, Victoria, Australia. He then went on to discuss the effects of radiation exchanges between parallel surfaces or closely spaced concentric cylinders.

E. M. Sparrow, of the University of Minnesota, Minneapolis, Minn., spoke on radiant emission, absorption, and transmission characteristics of cavities and passages. His talk summarized and generalized available information on emission and absorption characteristics of holes and cavities having various geometries.

In the next talk, W. M. Brandenberg and O. W. Clausen, of General Dynamics/Astronautics, San Diego, Calif., described an apparatus in which emittance measurements were made on flat samples as a function of the emission angle, temperature, and wavelength. These investigations, carried out in vacuum, were compared to theory. The talk concluded with an analysis of the errors associated with such investigations.

F. J. Kelly and D. G. Moore, of NBS, Washington, D.C., described an experimental test of analytical expressions for computing the thermal emittance of shallow cylindrical cavities. Measurements were made at room temperature with a cylindrical device having a movable plunger that permitted the length-to-diameter ratios of the cavity to be varied. Reflectance was meas-

ured and converted to emittance. The investigation showed that two of the theoretical expressions predicted the measured emittances to within close tolerances.

H. J. Keegan, J. C. Schleter, and V. R. Weidner, of NBS, described work involving attempts to correlate surface finish or texture with the diffuse spectral reflectance of metals. A variety of materials including aluminum, platinum, beryllium, steel, and rhodium-plated nickel is being prepared and evaluated in the investigation.

Further information regarding the effect of surface texture on diffuse spectral reflectance was provided in the final talk of the first session. D. B. Spangenberg, A. G. Strang, and J. L. Chamberlin, of NBS, defined explicitly the surface textures of the highly finished specimens employed in the measurements described in the immediately preceding talk. In many instances a single roughness parameter and a description of the finishing process is sufficient to describe a surface. However, in this work the surfaces are described in much more detail.

Session II

The second session consisted of a modified panel discussion with R. E. Gaumer, of Lockheed Missiles and Space Co., Palo Alto, Calif., acting as moderator. Each of the panel members presented a short discussion on the general subject of "Surface Effects." These discussions were followed by a number of invited comments by selected individuals. The discussion was then opened to the audience who submitted questions and comments to appropriate panel members. The panel consisted of H. H. Blau, Jr., Arthur D. Little, Inc.; G. Hass, Engineering Research and Development, U.S. Army; H. E. Bennett, U.S. Naval Ordnance Test Station, China Lake, Calif.; J. C. Richmond, NBS; and G. B. Heller, NASA Marshall Space Flight Center.

Session III

Chairman for the third session of the Symposium, entitled "Measurement Techniques," was C. P. Butler, of USNRDL. W. A. Clayton, of the Boeing Company, opened the session with comments on measurement techniques. These remarks pointed out the need for increasing the capability for measuring thermal radiation properties of materials. He indicated that the development of such measurements should be of a general nature rather than merely an ability to satisfy a particular purpose or an immediate need.

The second talk of the third session was presented by R. Stair and W. E. Schneider, of NBS. Their topic for discussion was "Standards, Sources, and Detectors in Radiation Measurement." They described the physical laws on which radiation measurements are based. They then discussed standards and methods for making the measurements, and the precision and accuracy obtained.

P. E. Schumacher, of Rocketdyne, a division of North American Aviation, Inc., described a circular aperture blackbody radiation source. The unit, designed for field calibration of missile- and air-borne radiometers, is self-contained with the exception of the electrical power source. It is usable over a broad spectral range at temperatures as high as 2300 °C.

A description of a rotating cylinder apparatus for determining the total normal emittance and normal spectral emittance of ceramic oxides was given by H. E. Clark and D. G. Moore, of NBS. This equipment operates in the spectral range of 1 to 15 μ at temperatures of 1200 to 1800 °C.

The next contribution was from R. G. Wilson, of the NASA Langley Research Center. He explained his work concerning emittance and reflectance measurements on materials employed in nose cones and other vehicles that may present ablation problems upon reentry into the Earth's atmosphere. These measurements were made at temperatures of from 1650 to 3300 °C.

Experiments for measuring the radiance ratio of optical specimens from 2.5 to 15 μ at temperatures of 600 to 1300 °K were conducted and the results reported by W. B. Fussel and R. Stair, of NBS. They developed equipment for measuring the optical properties of materials important in the design of infrared detection systems with acceptable precision.

N. J. Douglas, of Lockheed Missiles and Space Co., next spoke on directional solar absorptance measurements. He presented a method for determining the solar absorptance of thermal control surfaces from a knowledge of both the ratio of solar absorptance to the thermal emittance, and the emittance alone. He further reported the techniques, results, and errors associated with directional solar absorptance measurements.

The next presentation was given by G. W. McIntyre, of the Boeing Co. This report concerned the thermal characteristics, heat transfer, electric phenomena, and mathematics involved in obtaining thermal emittance measurements on electrical wire insulation. These parameters were determined in such a manner that a computer could perform the necessary calculations.

The final talk of this session was presented by O. Makarounis, of USNRDL. He presented an analysis of the conductive heat losses along the supporting wires of a specimen and the conductive air losses in a low temperature, evacuated chamber. Corrections for such losses, for total hemispherical emittance measurements determined by the decay method, in the 123 to 727 °K temperature range, were discussed.

Session IV

Chairman for Session IV, "Space Environmental Effects," was G. B. Heller, of the NASA Marshall Space Flight Center. The first presentation was given by J. E. Gilligan and R. P. Caren, of Lockheed Missiles and Space Company. They described results of their investigations to determine the mechanisms by which thermal control surfaces degrade under ultraviolet and nuclear radiations. Most of these investigations were conducted on dielectric and semiconductor materials.

"Solar-Wind Bombardment of a Surface in Space" was the title of the next talk by G. K. Wehner, of Gen-

eral Mills' Electronics Division, Minneapolis, Minn. This discussion was concerned with the effects of elementary particle bombardment determined from astrophysical observations and from measurements made with the satellites Explorer X, Lunik II, and Mariner II.

Similar problems were discussed by the next speaker, R. B. Merrill, of NASA Marshall Space Flight Center. He described an analytical model developed to predict the change in emittance from a surface caused by particle impact. This model compares favorably with experimental values.

In the next talk, J. A. Parker and C. B. Neel, of the NASA Ames Research Center, Moffett Field, Calif., and M. A. Golub, of the Stanford Research Institute, Menlo Park, Calif., spoke about laboratory and in-flight studies concerned with ultraviolet degradation of polymer films. Reflective white paints, consisting of inorganic pigments and organic polymeric binders, have been employed as thermal control surfaces for space vehicles. These studies show that the inorganic pigments are relatively stable, but that the organic portion of the films degrades when exposed to gamma and ultraviolet irradiation.

The next contribution was given by G. A. Zerlaut, Y. Harada, and E. H. Tompkins, of the Illinois Institute of Technology Research Institute. These investigators examined the ability of potential spacecraft coatings to withstand environmental space conditions. Zinc oxide and zinc sulfide were found to be unusually stable white pigments which form relatively stable coatings when dispersed in pure potassium silicate or in experimental methyl silicone polymers.

R. L. Olson, L. A. McKellar, and J. V. Stewart, of the Lockheed Missile and Space Company, spoke of their work in evaluating and predicting the operational behavior of selected materials. In this work, accelerated test methods were developed for materials exposed to ultraviolet radiation under vacuum conditions.

The final talk of this session was given by J. C. Arvesen, C. B. Neel, and C. C. Shaw. They reported preliminary results from a roundrobin testing program that was organized in November 1962, in an attempt to advance the technology of ultraviolet testing of thermal control coatings.

Session V

The final session, "Applications," was chaired by A. J. Katz, of the Grumman Aircraft Engineering Corp. Leading off this session was Dr. H. Tabor, Director of the National Physical Laboratory of Israel, who described their work in which thin film techniques were used to produce surfaces having high ratios of solar absorptance to hemispherical emittance. The theoretical basis for matching the optical properties of coating and substrate to produce blooming was described in detail.

D. F. Stevison and M. L. Minges, of the Air Force Materials Laboratory, considered some of the fundamentals relating to thermal energy transport in solids, then considered high-temperature measurement methods as applied to thermal optical property determinations.

W. Snoddy and E. Miller, of NASA Marshall Space Flight Center, surveyed the absorptance and emittance characteristics of several coating materials. The speakers cited, from results of a literature survey, areas where more research on thermal radiation is needed immediately, and areas where such research would be desirable for future use.

"Emittance Measurements of Solids Above 2000 °C" was the subject of the next talk presented by T. S. Laszlo, R. E. Gannon, and P. J. Sheehan, of the Avco Corp. They described the mathematical relationships, instrumentation, and techniques necessary for high-temperature emittance measurements with a calibrated solar furnace.

The next talk by R. P. Caren, A. S. Gilcrest, and C. A. Zierman, of the Lockheed Missiles and Space Co., described thermal radiation problems associated with evacuated cryogenic space chambers. Radiant energy absorptance, emittance, and reflectance of the surfaces of the vessel must be known accurately before the thermal radiation characteristics of a specimen within the chamber can be evaluated.

R. N. Schmidt and J. E. Janssen, of the Minneapolis-Honeywell Research Center, spoke on "Selective Coatings for Vacuum Stable High Temperature Solar Absorbers." These studies are basic to the development of thermal energy converters on spacecraft.

E. R. Streed and C. M. Beveridge, of the Philco Corporation, discussed problems associated with prelaunch and in-flight environments on low solar absorptance coatings. These investigations showed that ultraviolet radiation causes the greatest potential damage to such coatings.

C. H. Liebert and R. R. Hibbard, of the NASA Lewis Research Center, discussed their work in determining the effectiveness of solar reflectors in reducing heat transfer through the insulation of cryogenic fuel tanks located in space. These analyses were conducted with liquid hydrogen and oxygen stored in tanks at environmental locations equivalent to those of Mercury, Earth, and Mars.

The final talk of the symposium was presented by J. C. Arvesen of the NASA Ames Research Center. He discussed his investigations which showed that radiation shields having the proper configuration can effectively control space capsule temperature.

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[1] The Proceedings of the Symposium on the Thermal Radiation Properties of Solids *will be published in the near future and will be sent without charge to all registered participants. For other persons who wish to purchase a copy of the* Proceedings, *a nominal charge will be made. Inquiries concerning the* Proceedings *should be directed to Dr. S. Katzoff, National Aeronautics and Space Administration, Langley Research Center, Langley Field, Va.*

[2] *The most recent previous symposium in this field,* Measurement of Thermal Radiation Properties of Solids, *was held in Dayton, Ohio, in Sept. 1962. Proceedings of this symposium are for sale as* NASA Special Publication SP-31 *by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402, for a price of $3.50 (paper covers).*

Five Changes in NBS Radio Broadcasts

A number of changes in the schedules and contents of NBS radio broadcasts of time and frequency standards have been made recently. These changes are:

(1)* At zero hours Universal Time 1 April 1964 the transmitting clocks controlling NBS stations WWV, WWVH, and WWVB were retarded 100 milliseconds. The last such adjustment, necessitated by changes in the speed of the earth's rotation, was made on 1 November 1963. U.S. Navy stations NBA, NPG, NPM, NPN, and NSS were also retarded at the same time.

(2) On 1 July 1964 stations WWVB and WWVL will resume continuous service except for the biweekly silent periods previously announced (1300 UT Tuesday to 0100 UT Wednesday; the stations alternate operation on successive Tuesdays).

(3) WWV now broadcasts (service started Nov. 15, 1963) the frequency offset—M150—in Morse code immediately following the "on-the-hour" voice announcement, and broadcasts propagation forecasts in Morse code every five minutes following code time announcements. The M150 symbol indicates the signal as broadcast is offset from the U.S. Frequency Standard by minus 150 parts in 10^{10}.

(4) A new schedule of geophysical alerts on WWV and WWVH was initiated on 1 April 1964. The following signals are broadcast in Morse code (7 words/min) on WWV during the first half of the 19th minute and on WWVH during the first half of the 49th minute past each hour:

GEO–MMMMM (Magnetic storm)
GEO–NNNNN (Magnetic quiet)
GEO–CCCCC (Cosmic ray event)
GEO–SSSSS (Solar activity)
GEO–QQQQQ (Solar quiet)
GEO–WWWWW (Stratospheric warming)
GEO–EEEEE (No geoalert issued)

(5) By agreement with the Naval Observatory WWV and WWVH began broadcasting on May 1, 1964, daily corrections to the regular time signals to enable users to obtain a very accurate value of UT–2. During the last half of the 19th minute of each hour on WWV and the last half of the 49th minute of each hour on WWVH Morse code signals are broadcast as follows:

UT2 space AD or SU space three digits.

UT–2 is obtained by adding or subtracting (as indicated) the number of milliseconds indicated by the last three digits to the time as broadcast. The symbols will be revised on a daily basis, the new value appearing for the first time during the hour after midnight UT, and continuing for the following 24-hr period.

*This adjustment was also announced jointly with Naval Observatory on March 9, 1964.

MECHANIZED DOCUMENTATION SYMPOSIUM

A Symposium on Statistical Association Methods for Mechanized Documentation, held March 17 to 19 in Washington, D.C., attracted to its sessions over 200 specialists on data handling. They met to discuss ways of cataloging and recalling data by the use of machines in place of human operators. The Symposium was sponsored by the National Bureau of Standards Institute for Applied Technology and the American Documentation Institute; its sessions were held at the Smithsonian Institution in Washington.

The Symposium was opened on March 17 with remarks by Dr. Donald A. Schon, Director of the NBS Institute for Applied Technology, in which there are many NBS programs utilizing mechanized information retrieval techniques. The sessions that followed included talks and panel discussions on many aspects of handling documents automatically—their categorization, indexing, and retrieval—which should prove to be of value to the scientific community in years to come.

The recent proliferation of scientific knowledge and publication has encouraged the development of electronic methods of storage and retrieval. The next step, automatic indexing, was the topic of many of the papers and panel comments at the Symposium. Particular emphasis was given to computational techniques for determining coefficients of correlation and other measures of statistical association among text words, indexing terms, terms, and documents. Data-processing technologists interested in these subjects will find information on them in the Symposium Proceedings, to be published by the U.S. Government Printing Office later this year.

Shock-Tube Kinetic Studies Using a Reference Standard

A COMPARATIVE technique in which the reaction rate of a gas is found by heating it in a shock tube [1] with a gas whose rate is known has been developed by Dr. Wing Tsang of the NBS Institute for Basic Standards. With this technique, using isopropyl bromide as the "standard," rate constants for *tert*-butyl chloride and bromide have been determined that agree with earlier static determinations. These preliminary experiments, which prove the validity of the technique, should lead to shock-tube determination of the rate constants for the decomposition of other interesting compounds.

The shock tube offers several advantages for the determination of kinetic data. One advantage is that high temperatures are easily attained; another is that homogeneous heating and short reaction times eliminate wall effects. Also, the short heating time and the low concentrations of the sample minimize chain reactions. One limitation of the shock-tube technique, however, has been the difficulty involved in the accurate determination of the temperature associated with the shock. The comparative technique developed by NBS avoids this difficulty completely, as the use of a standard gas whose rate constant is known eliminates the need to determine the temperature. Further, as the standard and test gas are exposed simultaneously, there is large-scale cancellation of errors associated with other reaction parameters.

The brass shock tube used in this work is 1.0 in. I.D. and 10.5 ft long. A cellophane diaphragm confines the driver gas (either helium or hydrogen, depending on the temperature desired) to 5 ft at the left end of the tube until the diaphragm is ruptured by a solenoid-driven plunger. A 36-liter argon-filled dump tank, positioned 1.5 in. to the right of the diaphragm, serves to damp out shock waves that are reflected from end to end during a run. Such damping is necessary to prevent reheating of the sample. The response of a quartz pressure transducer near the right end of the tube is used to determine the time over which the sample is heated. A sampling port ½ in. from the right end is used to withdraw samples for analysis.

Isopropyl bromide, whose rate constant has been accurately determined by several different methods, was used as the reference material in this particular investigation. Runs were made by admitting mixtures of the reference gas and either *tert*-butyl chloride or *tert*-butyl bromide in argon to the tube and opening the valve to the dump tank. The argon in the dump tank was brought to the pressure in the tube before opening the valve. The driver gas was then admitted, the diaphragm ruptured, and a sample of the gas taken for analysis. The procedure was carried out as rapidly as possible to minimize diffusion effects.

Because both the standard and the test gas decompose through unimolecular dehydrohalogenation,

$$C_3H_7Br \rightarrow C_3H_6 + HBr \text{ and } C_4H_9X \rightarrow C_4H_8 + HX,$$

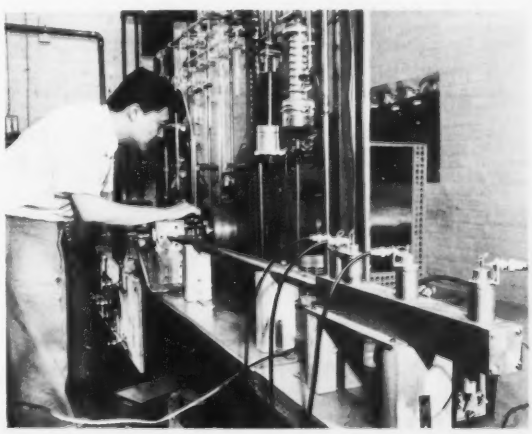
gas chromatographic analysis of the organic products of decomposition gives the percentage reaction for each of the gases. As the rate constant k of the iospropyl bromide is known, the constant for the gas under investigation can be calculated from the reaction time and the relative amounts of decomposition. The results of runs at different temperatures are:

$$k(C_4H_9Cl) = 10^{13.74}e^{-44,690/RT}(sec^{-1}) \text{ and}$$
$$k(C_4H_9Br) = 10^{13.87}e^{-41,490/RT}(sec^{-1}),$$

figures which compare well with earlier static determinations at lower temperatures. The results of duplicate runs show good precision.

[1] Comparative rate measurements with a single-pulse shock tube, *by W. Tsang, J. Chem. Phys.* **4**, *1171 (1964)*.

A technique in which the reaction rate of a gas is found by heating it in a shock tube with a gas whose rate is known was developed by Dr. Wing Tsang. Using isopropyl bromide as a standard, Dr. Tsang, here adjusting the apparatus, has determined rate constants of *tert*-butyl chloride and bromide that agree well with static results.



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CRPL Ionospheric Predictions for August 1964. Three months in advance. Number 17, issued May 1964. 15 cents. Annual subscription: $1.50; 50 cents additional for foreign mailing. Available on a 1-, 2-, or 3-year subscription basis.

Journal of Research of the National Bureau of Standards.

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Section B. Mathematics and Mathematical Physics. Issued quarterly. Annual subscription: Domestic, $2.25; foreign $2.75; single copy, 75 cents.

Section C. Engineering and Instrumentation. Issued quarterly. Annual subscription: Domestic, $2.25; foreign, $2.75; single copy, 75 cents.

Section D. Radio Science. Issued monthly. Annual subscription: Domestic, $9; foreign, $11.50; single copy, $1.00.

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J. Res. NBS **68A** *(Phys. and Chem.), No. 3 (May–June 1964).*

Compressibility of natural rubber at pressures below 500 kg/cm^2. L. A. Wood and G. M. Martin,

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Crystallization of anhydrous copper sulfate from sulfuric acid-ammonium sulfate mixtures. P. M. Gruzensky.

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Electrical properties of sea ice at 0.1 to 30 Mc/s. F. L. Wentworth and M. Cohn.

ELF and VLF waves below an inhomogeneous anisotropic ionosphere. J. Galejs.

The effects of a small local change in phase velocity on the propagation of a VLF radio signal. D. D. Crombie.

Auroral-zone absorption effects on an HF arctic propagation path. R. D. Hunsucker.

Amplitude-probability distribution of atmospheric radio noise. P. Beckmann.

An approximate method for computing diffraction patterns caused by ionospheric irregularities. R. Gagnon.

Wave propagation in stratified random media. Y. M. Chen.

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Determinations based on duplication of readings. J. A. Speckman.

General application of Youden's rank sum test for outliers and tables of one-sided percentage points. T. A. Willke.

A generalization of Rennie's inequality. A. J. Goldman.

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J. Res. NBS **68C** *(Eng. and Instr.), No. 2 (Apr.–June 1964).*

Fringe pattern of an oscillating Fabry-Perot interferometer. K. D. Mielenz.

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